

Enrichment of decanoic acid in cuphea fatty acids by molecular distillation[☆]

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Abstract

The introduction of a new crop often requires the development of new products and purification techniques of either the oil or fatty acids. Most new crops enter the cosmetic market first due to their high rates of return. However, the cosmetic market often demands highly pure and colorless materials. A molecular distillation unit is a powerful method of separation; separation occurs at extremely low pressures, and therefore at reduced temperatures compared with normal distillations and makes it very suitable for cuphea germplasm line PSR-23 (*C. lanceolata* × *C. viscosissima*) fatty acids. Refined cuphea fatty acids, which are mainly short-chain saturated fatty acids such as decanoic acid (C-10), were distilled using a lab-scale centrifugal molecular distillation unit to yield a distillate that was colorless (Gardner color = 1–). The optimal distillation conditions were explored by varying the rotor temperature and flow rate onto the rotor. As the conditions were varied, the distillate and residue were monitored for Gardner color, fatty acid composition, and mass split rate between the distillate and residue. Under a high flow rate (2.23 g/min), enrichment of C-8 and C-10 in the distillate was increased to 96% in a single-pass distillation (rotor temperature = 65 °C) with no unsaturation in the distillate while maintaining a split ratio of 1.5. A high flow, multiple-pass distillation (rotor temperature = 70 °C) of a distillate fraction produced a distillate that was 99.8% saturated fatty acid and 0.2% unsaturated with very high split flow ratios. Overall, laboratory distillation of cuphea fatty acids successfully demonstrated the ability to achieve either high quantities of C-8 and C-10 or total saturated samples.

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1. Introduction

Coconut (*Cocos nucifera*) and palm kernel (*Elaeis guineensis*) oils provided more than 450 thousand tons

of decanoic acid to the global market in 2006. The oils were in wood preservatives (Yoshida and Inuma, 2004), activity against termites (Goettsche and Borck, 1994), dental compositions (Velamakanni et al., 2006), lubricants (Cermak and Isbell, 2004), fabric softeners (Hohener and Frick, 2003), cosmetic agents for hair (Hoppe and Engel, 1989), and other cosmetics (Ishii and Mikami, 1995).

At relatively low temperatures, cuts of saturated fatty acids can be successfully distilled from coconut and palm kernel fatty acids to produce nearly colorless fatty acids. There are many compounds that are sensitive to heat

[☆] Names are necessary to report factually on available data; however, the USDA neither guarantees nor warrants the standard of the product, and the use of the name by USDA implies no approval of the product to the exclusion of others that may also be suitable.

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such as high vacuum oils (Rees, 1980), vegetable oils (Cermak and Isbell, 2002; Isbell and Cermak, 2004), pharmaceuticals, and cosmetics (Batistella and Maciel, 1996) that prohibits the use of conventional distillations. Molecular or short path distillation has been known for sometime (Biehler et al., 1949), where a high vacuum is used to achieve distillation of thermally unstable materials; this method is often the most economically feasible method of purification. Centrifugal and falling films are two basic types of molecular distillation units that use a short exposure of the distilled liquid to the evaporating cylinder. The high temperature exposure time in these distillation units is on the order of a few seconds to tenths of a second as the liquid is spread evenly in the form of a film (Micov et al., 1997). These types of distillation units have been used successfully to demonstrate and compare the distillation of many different compounds, such as carotenoids from palm oil (Batistella and Maciel, 1998) and estolides (Isbell and Cermak, 2004).

Cuphea is an annual plant that produces a small seed with oil that is rich in saturated medium-chain triacylglycerols. The initial oil characterization of a number of *Cuphea* species (*Lythraceae*) was done at the U.S. Department of Agriculture (USDA) Research Center in Peoria, IL, in the early 1960s (Miller et al., 1964). With the need for higher seed yields, oil content, and less seed shattering, Steve Knapp (1993) at Oregon State University began developing promising cuphea crosses. One of these new germplasm lines, PSR-23 (an interspecific hybrid between *C. lanceolata* and *C. viscosissima*), with partial seed retention, was developed at the University of Oregon and was planted in the Midwest and mechanically harvested and dried (Cermak et al., 2005) by researchers at USDA in Morris, MN, and Peoria, IL since 2000. PSR-23 is a germplasm line that is high in C-10, decanoic acid (Table 1).

Many *Cuphea* species contain octanoic acid as the major fatty acid. Obtaining the correct *Cuphea* species or cuphea line with the highest levels of decanoic acid may provide a strong economic incentive for incorporating cuphea as a new rotation crop. Once cropping practices have been successfully and economically

developed for cuphea, cuphea would serve as a domestic source of decanoic acid and would lessen USA's demand for foreign imports of coconut and palm kernel oils.

Estolides have been previously synthesized by USDA's National Laboratory in Peoria, IL from C-10 decanoic acid (Cermak and Isbell, 2004). These estolides have useful properties as lubricants based on the physical properties exhibited by these C-10 materials. The one problem associated with the decanoic estolides is the color of the final product. Commercial decanoic acid is not as refined as other mid-chain fatty acids, so that removal of the color bodies is necessary for light colored estolides. Lubricant manufacturers and consumers would prefer lubricants with Gardner colors similar to current petroleum oils. To obtain these properties, the starting material must be distilled, which is costly and can lead to undesired additional color bodies with certain distillation techniques. The ability to achieve a fast and mild distillation of short-chain fatty acids could be used to obtain oil with low Gardner color while the residue would not be darkened by the distillation process. These improvements with decanoic acid would help with the commercialization of cuphea as a new oil seed and give Midwest farmers a valuable rotation crop.

The objective of this study was to investigate the general conditions necessary for laboratory centrifugal distillation of decanoic acid from cuphea fatty acids. Fatty acid profiles, Gardner colors, and flow rate requirements were examined to determine the best set of operating conditions.

2. Materials and methods

2.1. Materials

Cuphea seeds were harvested from USDA plots in Morris, MN. The fatty acid methyl ester (FAME) standard mixtures were obtained from Alltech Associates, Inc. (Deerfield, IL) and NuCheck (Elysian, MN). Concentrated sulfuric acid was obtained from T.J. Baker Chemical Co. (Clifton, NJ). Methanol and hexanes were

Table 1
Refined cuphea fatty acid composition

	Fatty acid (%) ^a							
	8:0	10:0	12:0	14:0	16:0	18:0	18:1	18:2
Cuphea ^b	0.8	81.9	3.2	4.3	3.7	0.3	3.6	2.0

Determined by GC (SP-2380, 30 m × 0.25 mm i.d.).

^a Standard deviation < ±0.10.

^b Germplasm line PSR-23.

obtained from Fisher Scientific Co. (Fairlawn, NJ). Ethanol was obtained from Aaper (Shelbyville, KY).

2.2. Cuphea seed press, refining, and hydrolysis

Clean, whole Cuphea seeds (9.7% moisture content) were milled using a pair of smooth rolls (Model SP900-12 Roller Mill, Roskamp Champiom, Waterloo, IA) with gap between the rollers set to 0.25 mm. The milled seeds were immediately loaded into a preheated seed cooker (Laboratory Seed Cooker/Conditioner Model 324, French Oil Mill Machinery Company, Piqua, OH). The seed temperature was monitored so as not to exceed 93 °C during cooking. Cooking time was 75 min and the final moisture content of the cooked milled seed was 5.9% (dry based). The cooked milled seeds were screw pressed using a Heavy Duty Laboratory Screw Press (Model L 250, French Oil Mill Machinery Company, Piqua, OH).

The filtered crude oil was acid degummed to remove the phosphatides. The free fatty acids were removed by neutralizing the degummed oil with 14.3% NaOH and the resulting soapstock was separated by centrifugation. The refined oil was bleached using 4.5% bleaching clay (Tonsill 167 FF, Sud-Chemie Adssrbents, Inc., Meigs, GA) and 2% activated carbon (Darco KB, Norit Americas, Inc., Marshall, TX). The bleached oil was deodorized for 2 h at 260 °C with sparge steam at 1–2% w/w per hour to yield refined cuphea seed oil.

The refined cuphea seed oil (350 g, 559 mmol) was hydrolyzed by the addition of 2.0M KOH/EtOH followed by heating to reflux for 60 min. After cooling to room temperature, the reaction mixture was placed in a 6l Erlenmeyer with 1l of hexane and cooled in an ice bath. A 1 M HCl solution was slowly added to the cooled hydrolysis mixture with overhead stirring. Addition was maintained until the solution was slightly acidic, as measured by pH paper. The mixture formed an emulsion as the transition occurred. The pH of the organic layer was adjusted to 5.3–6.0 with the aid of a pH 5 buffer (NaH₂PO₄, 519 g in 4l H₂O, 2 (100 ml). The organic layer was removed, dried over sodium sulfate, and filtered. All reactions were concentrated in vacuo then kugelrohr-distilled at 160–190 °C at 0.013–0.067 kPa to purify the fatty acids (331.3 g). A small sample of the fatty acids was then esterified to the corresponding methyl ester under conditions described above to yield the cuphea fatty acid profile, Table 1.

2.3. Equipment and procedures

2.3.1. Gas chromatography (GC)

GC analysis was performed with a Hewlett-Packard 6890N Series gas chromatograph (Palo Alto, CA) equipped with a flame-ionization detector and an autosampler/injector. Analyses were conducted on a SP-2380 30 m × 0.25 mm i.d. column (Supelco, Bellefonte,

Table 2
Effects of molecular distillation parameters on short-chain fatty acids, split ratio, and color

Trial	Rotor temperature (°C)	Flow (g/min)	Split flow ^a	C-8 and C-10 fatty acids (%) ^b		Gardner color	
				Distillate ^c	Residue ^c	Distillate	Residue
1	40	1.49	0.03	98.9	81.9	1–	1
2	40	0.49	0.03	98.9	81.5	1–	1
3	50	1.36	0.30	98.5	77.4	1–	2
4	50	0.54	0.12	98.8	80.3	1–	3
5	55	1.65	0.93	97.0	69.5	1–	1
6	55	0.49	0.37	98.3	76.9	1–	1
7	60	1.99	1.30	96.5	63.4	1–	1
8	60	0.52	0.85	96.2	69.2	1–	2
9	65	2.23	1.47	95.5	62.7	1–	1
10	70	1.36	1.44	95.1	62.3	1–	3–
11	70	0.46	0.98	96.1	67.9	1–	2
12	90	1.72	1.79	88.2	58.9	1–	1
13	90	0.50	1.97	89.8	66.6	1–	1
14	110	1.87	4.63	83.8	69.1	1–	6–
15	110	0.54	4.92	83.3	66.9	1–	7

Heated rotor spinning at 28.75 Hz under a high vacuum (0.08–0.40 Pa).

^a Distillate to residue ratio.

^b Saturated fatty acids determined by GC (SP-2380, 30 m × 0.25 mm i.d.).

^c Standard deviation < ±0.10.

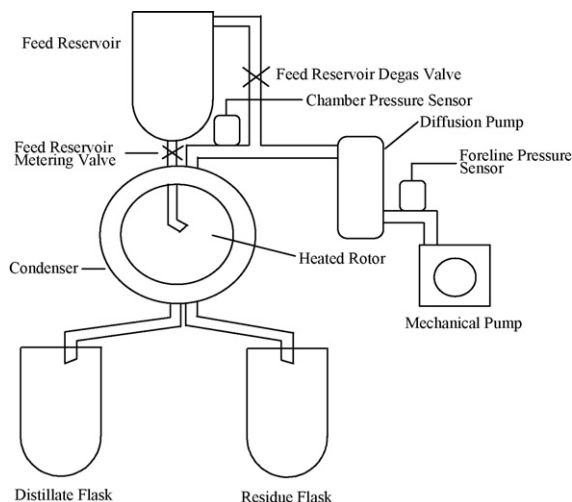


Fig. 1. Schematic diagram of Myers Lab 3 short path molecular distillation unit.

PA). Saturated C₈–C₃₀ FAMES provided standards for making fatty acid assignments.

Parameters for SP-2380 analysis were: column flow 1.4 ml/min with helium head pressure of 136 kPa; split ratio 10:1; programmed ramp 120–135 °C at 20 °C/min, 135 to 265 °C at 7 °C/min, hold 5 min at 265 °C; injector and detector temperatures set at 250 °C. Retention times for eluted peaks were: methyl octanate 2.97 min, methyl decanate 3.60 min, methyl dodecanate 4.59 min, methyl tetradecanate 5.96 min, methyl hexadecanate 7.59 min, methyl octadecanate 9.34 min, methyl oleate 9.86 min, methyl linoleate 10.65 min, and methyl linolenate 11.58 min.

2.3.2. Molecular distillations

Fractional distillations were performed using two different flow rates (high and low) and eight different rotor temperatures (Table 2) using a Myers Lab 3 short path molecular distillation unit (Myers Vacuum, Kittanning, PA). A diagram illustrating the operational features of this unit is presented in Fig. 1. The condenser temperature was set at 40 °C, rotor speed was constant at 1725 rpm, and cold tap water was used to cool the diffusion pump and rotor bearing. Vacuum was maintained at (0.08–0.40 Pa) at both the chamber and foreline pressure sensors (Fig. 1).

Fractionation of cuphea fatty acids into octanoic and decanoic acids (C-8 and C-10) versus higher molecular weight fatty acids was studied on a short path distillation unit. Feed stock was metered onto a heated rotor spinning at 28.75 Hz placed under a high vacuum (0.08–0.40 Pa) as shown in Fig. 1. Once on the hot spinning surface, a thin film was formed where rapid heat transfer occurred,

thus promoting distillation of the lower molecular weight fatty acids from the higher weight fatty acids. The material that remained undistilled flowed rapidly off the rotor into the residue flask, which was maintained under vacuum at room temperature. This short residence time at high temperature helps reduce degradation and color bodies of the residue unlike other distillations where the sample remains at the distillation temperature for several hours. The distillate fraction is collected on the front condenser plate, which is cooled with tempered water (40 °C to prevent solidification). The distillate exited the chamber and flowed into the distillate flask, which was also maintained under vacuum at room temperature.

2.3.3. Molecular distillation—sampling schedule

All data (Tables 2–4) were collected by setting the instrument to the desired conditions then allowing the instrument to reach equilibrium. Individual data points were collected over a given time (1–2.5 h depending on the flow rates). The flow rates were then calculated based on the mass of distillate and residue collected divided by the sample time.

2.3.4. Gardner color

Gardner color was measured on a Lovibond 3-Field Comparator from Tintometer Ltd. (Salisbury, England) using AOCS method Td 1a-64 (Firestone, 1994). Gardner color of both the residue and distillate materials was measured throughout the distillation process. The + and – notation was employed to designate samples that did not match one particular standard.

2.3.5. GC analysis fatty acids

Analytical samples for GC were prepared by heating a 10 mg sample of distillate or residue in 1 ml of 1M H₂SO₄/MeOH in a sealed vial, placed in a heating block and heated to reflux for 15 min. The solution was transferred to a separatory funnel with water (1 ml) and washed with hexanes (2 × 2 ml), dried over sodium sulfate, gravity filtered, placed in a GC vial, sealed, and injected into the GC.

2.3.6. Statistical analysis

The mean value of duplicate determinations for flow rate, % fatty acids, and split ratios were analyzed. Standard deviations for all the GC data was less than ±0.10. ORIGIN 7 software was used to make all graphs.

3. Results and discussion

The basic experimental conditions for the two different flow rates, high and low, over eight different

Table 3
Multiple-pass distillation of the residue sample at 55 °C

Trial	Distillation (pass)	Flow (g/min)	Split flow ^a	C-8 and C-10 fatty acids (%) ^b		Saturated fatty acids (%) ^b		Gardner color	
				Distillate ^d	Residue ^d	Distillate ^d	Residue ^d	Distillate	Residue
5 ^c	First	1.65	0.93	97.0	69.5	100	88.5	1–	1
16	Second	1.38	0.35	97.1	58.8	100	83.9	1–	NA
17	Third	1.48	0.09	97.2	54.9	100	82.2	1–	4+

Heated rotor spinning at 28.75 Hz under a high vacuum (0.08–0.40 Pa).

^a Distillate to residue ratio.

^b Saturated fatty acids determined by GC (SP-2380, 30 m × 0.25 mm i.d.).

^c See Table 2.

^d Standard deviation < ±0.10.

Table 4
Multiple-pass distillation of the distillate sample at 70 °C

Trial	Distillation (pass)	Flow (g/min)	Split flow ^a	C-8 and C-10 fatty acids (%) ^b		Saturated fatty acids (%) ^b		Gardner color	
				Distillate ^c	Residue ^c	Distillate ^c	Residue ^c	Distillate	Residue
18	First	1.71	2.50	91.0	57.4	98.5	81.1	1–	1
19	Second	2.06	11.33	92.4	69.8	99.1	88.5	1–	6+
20	Third	1.74	15.50	93.3	72.3	99.4	89.9	1–	13–
21	Fourth	1.84	16.59	93.7	81.9	99.8	94.2	1–	11–
22	Fifth	1.85	11.79	94.6	83.1	99.8	96.2	1–	4+

Heated rotor spinning at 28.75 Hz under a high vacuum (0.08–0.40 Pa).

^a Distillate to residue ratio.

^b Saturated fatty acids determined by GC (SP-2380, 30 m × 0.25 mm i.d.).

^c Standard deviation < ±0.10.

rotor temperature settings, their effects on split ratio determined by mass, Gardner colors, and percent short saturated fatty acids are listed in Table 2. The split ratios of the high and low flow rates across the rotor are similar (Fig. 2). However, as the rotor temperature is increased to 110 °C and greater, the split ratios increased with the lower flow rate. The increased mass in the distillate fraction would be expected at the lower flow rate

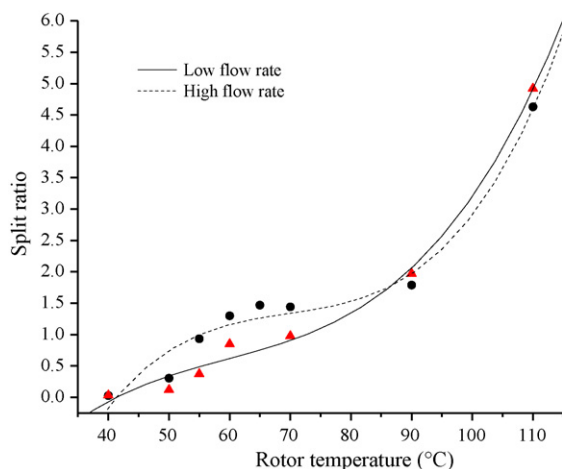


Fig. 2. Effect of rotor temperature on split ratio (distillate to residue).

because heat transfer to a smaller amount of material passing across the rotor would be more efficient (Isbell and Cermak, 2004). Data points are not reported past 110 °C because the saturated fatty acids in this study were well distilled before that temperature.

As the rotor temperature was increased the split ratios of distillate to residue increased and affected what was being distilled over. At increased temperatures, more unsaturates and higher chain saturated fatty acids were distilled affecting the purity of the sample. The purity of the distillate as defined by the percent of octanoic (C-8) and decanoic (C-10) fatty acids is shown in Fig. 3. Both the high and low flow rates gave similar results as the temperature was increased. All temperatures 70 °C and less provided materials that were greater than 95% enriched in C-8 and C-10. With a lower rotor temperature a greater quantity of short saturated fatty acids was present in the distillate fraction. As the rotor temperature was increased, both the low and high flow rates distillates contained lower percentages of the short saturated fatty acids, but the amounts of distillate increased over the amount of residue.

The amount of total saturates in the distillates as the rotor temperature was increased at the high flow rate is shown in Fig. 4. The distillate fractions did not contain

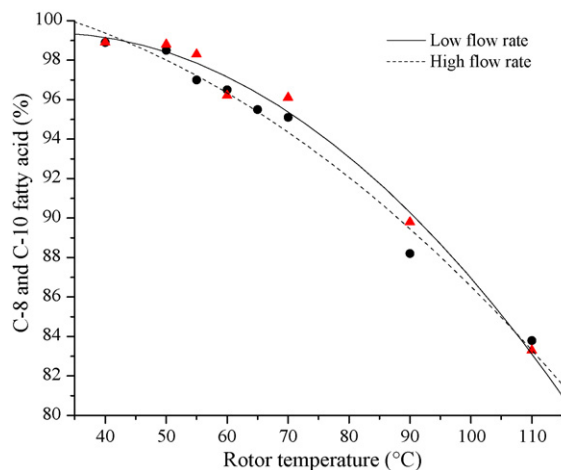


Fig. 3. Effect of rotor temperature the amount of C-8 and C-10 saturated fatty acid in the distillate.

any unsaturation until the rotor temperature was greater than 65 °C. The same results were observed for the low flow rate distillate fractions. The key to short-chain cuphea fatty acid distillation was to obtain conditions that allowed for optimal flow rates while achieving the best percent of short-chain fatty acids or total saturated fatty acids.

Single-pass distillation is the simplest and least expensive mode of distillation, but some applications require additional distillations to achieve difficult separations. Separating materials that are very close in weight or have very similar boiling points can require multiple-pass distillations. The multiple-pass distillation of the high flow rate residue sample at 55 °C is shown in Table 3. The first distillation at 55 °C achieved a 0.93 distillate ratio (Table 3). As the residue fraction was re-

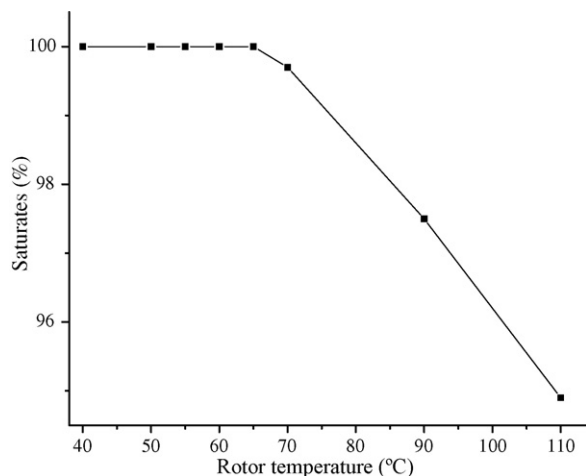


Fig. 4. Effect of rotor temperature on the amount of saturated fatty acid in the distillate (high flow rate).

distilled under similar conditions, the second and third re-distillations had lower split flow ratios, distillate to residues of 0.35 and 0.09, respectively. The lower split flow rates were expected because there was less light weight saturates in each consecutive distillation sample leading to a “heavy” distillation sample, i.e., less material that would distill under these conditions. However, the percent of C-8 and C-10 as well as the percent saturated fatty acids either increased or remained at 100% (respectively) with each multiple-pass. The residues from these re-distillations showed the expected decrease in the percent of C-8 and C-10 and overall saturates with each multiple-pass.

As the rotor temperature was increased, the distillate to residue split ratio increased, but the percent of short-chain fatty acids decreased in the distillate samples. The effects of multiple-pass distillation of the distillate fraction at 70 °C with a high flow rate are shown in Table 4. The split ratios of the multiple-pass distillations increased with each consecutive distillation from 2.50 (Table 4, Trial 18) to 16.59 (Table 4, Trial 21). The distillate percents of C-8 and C-10 and overall saturates all increased as expected (Table 5) as the distillate was further purified to obtain high percents of total saturates 99.8% (Table 4, Trial 21). The fatty acid profile for the multiple-pass distillation is shown in Table 5. After the first pass (Trial 18), stearic and linolenic fatty acids were removed and each additional pass started to remove the linoleic and oleic fatty acids. The final pass yielded a material that contained only 0.2% oleic as the only unsaturation in the fraction.

One of the main reasons for performing single or multiple-pass distillations was to aid in the removal of color bodies. All the distillates had very excellent Gardner colors 1–, which is the lowest color rating (Tables 2–4). The residues varied in the amount of color bodies in the samples depending on whether the residue was a single or multiple-pass distillation. A multiple-pass distillation of a distillate is prime example to explore the removal of color bodies. The first distillation gave a distillate (Table 4, Trial 18) with a Gardner color of 1–, however, when that material was re-distilled, the residue led to a Gardner color of 6+ (Table 4, Trial 19). Each consecutive distillation had higher split flow rates and showed that most of the material was being distilled. This increase was expected as only small amounts of saturates and/or color bodies were left to be removed with each distillation. At Trial 20, the color bodies peaked (Gardner color 13–) and the Gardner color value started to decrease as the color was being completely removed from the sample. This multiple-pass distillate would be very desirable for very color sensitive applications.

Table 5

Fatty acid profile of distillate distillations at 70 °C

Trial	Distillation (pass) ^a	Fatty acid (%) ^b								
		8:0	10:0	12:0	14:0	16:0	18:0	18:1	18:2	18:3
18	First	0.8	89.8	3.1	3.0	1.6	0.0	1.0	0.6	0.0
19	Second	0.7	91.4	3.0	2.6	1.2	0.0	0.6	0.4	0.0
20	Third	0.7	92.4	2.9	2.4	0.9	0.0	0.4	0.3	0.0
21	Fourth	0.6	92.9	2.9	2.2	0.8	0.0	0.4	0.2	0.0
22	Fifth	0.6	93.8	2.7	2.0	0.6	0.0	0.2	0.0	0.0

Determined by GC (SP-2380, 30 m × 0.25 mm i.d.).

^a Heated rotor spinning at 28.75 Hz under a high vacuum (0.08–0.40 Pa).^b Standard deviation < ±0.10.

With the Myers 3 being a laboratory molecular unit, one of the main goals was to have a high throughput while collecting saturated quality material. This equipment would meet these high throughput conditions when the rotor temperature was set to 65 °C with a high flow rate of 2.2 g/min while maintaining a distillate to residue split ratio of about 1.5 as demonstrated in Table 2 (Trial 9) and Fig. 4. The distillate produced would contain high percents of C-8 and C-10 as well as no unsaturates. The Gardner color values were excellent under these conditions as well.

4. Conclusions

Cuphea fatty acids can be effectively separated with a centrifugal molecular distillation unit such as the Myers 3 lab-scale. The precise distillation conditions were determined by varying the conditions to obtain distillate that was light in color, Gardner color = 1– which is ideal for cosmetic applications, to obtain distillate that was very high in octanoic (C-8) and decanoic (C-10) fatty acids, and 100% saturated fatty acids. Conditions were determined for the distillation of saturates from the unsaturates and fatty acid profiles were reported. The rotor temperature and flow rate had the greatest impact on the Gardner color and the fatty acid composition of the distillate.

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